(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
15 November 2001 (15.11.2001)

PCT

(10) International Publication Number
WO 01/85879 A1

(51) International Patent Classification[7]: C10M 125/26, 169/04

(21) International Application Number: PCT/US01/14430

(22) International Filing Date: 4 May 2001 (04.05.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/565,961 5 May 2000 (05.05.2000) US

(71) Applicant and
(72) Inventor: BACARELLA, Robert [US/US]; 150 Davis Street, Hauppauge, NY 11788 (US).

(74) Agent: SHEA, Timothy; Bowditch & Dewey, LLP, 161 Worcester Road, Framingham, MA 01701 (US).

(81) Designated States *(national)*: AU, BR, BZ, CA, CN, CR, CZ, DE, DE (utility model), GB, ID, IL, JP, KP, KR, MX, NO, NZ, RU, SG, SK, VN, ZA.

(84) Designated States *(regional)*: Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR).

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: HIGH-TEMPERATURE SYNTHETIC LUBRICIOUS COMPOSITION

(57) Abstract: Synthetic lubricant compositions and methods for their manufacture and use are provided. The compositions comprise mixtures of hydrogenated poly-α-olefins, styrene-ethylene/propylene copolymer, petroleum hydrocarbons; fumed silica, propylene glycol, and PTFE.

synthetic multi-purpose, heavy duty grease specifications

Composition		Synthetic / Polymer
Visual Appearance		Optically Clear
Shelf Life		> 10 years
Oil Separation Test	ASTM D1742 60°C 24 Hours	Zero
	120°C 24 Hours	Zero
Viscosity	Brookfield at 25°C	1800 mcps +/- 100mcps
Cone Penetration	ASTM D217 (unworked)	
	at +25°C	240 +/- 20
	at -20°C	160 +/- 20
Drop Test	ASTM D566 at 250°C	Zero
Flash Point	ASTM D92	180°C
Continuous Use Temp.	Grease remains flexible & stable	-41°C to 204°C
Effect on Copper	ASTM D1261	Zero
Effect on plastic, steel iron, bronze, acrylate	Electron Microscopy Detection of deterioration	No evidence is observed
Volatility	150°C for 24 hours	< 1%
Evaporation Test	ASTM D972 85°C for 24 hrs.	< .3%
Oxidation Induction Time	Bellcore TR- TSY000421	> 20 Minutes
Water Content	Karl Fisher Method	< .001%
Hydrogen Generation	ASTM D1018	< 30 ppm
Four Ball Wear Test	ASTM D2266 Scar diameter	0.48 mm
Salt Spray Test	ASTM B117	Pass
Four Ball E.P. Test	ASTM D2596	
	Load Wear Index	43.25 kg.
	Weld Point	208 kg.
Water Washout	ASTM D1264	
	37.8°C (100°F)	< 1%
	79.4°C (175°F)	< 1%
Rust Preventative Test	ASTM D1743	Pass

WO 01/85879 A1

HIGH-TEMPERATURE SYNTHETIC LUBRICIOUS COMPOSITION

Field of the Invention

This invention relates to high-temperature synthetic lubricious compositions that are suitable for use in operations including the processing and preparation of foodstuffs.

Background of the Invention

A wide variety of industrial and mechanical apparati require lubrication of their moving parts to prevent premature wear and failure of the equipment. Natural lubricants, such as animal and vegetable fats, oils, and greases have been known for millennia. However, as the industrial age arrived, these were found not to be suitable in the high temperature, high pressure conditions experienced in the machinery being developed. Petroleum-based lubricants have been known for over a century and have been developed for many such applications. However, the cost of locating, extracting, and refining crude oil to manufacture these lubricants, as well as limitations on their performance and durability, led to the development in this century of synthetic lubricants.

Synthetic lubricants have been developed to meet a variety of needs and have in the last twenty years gained in both their technical performance and their popularity. However, most synthetic lubricants include ingredients that are not suitable for certain applications, *e.g.*, for use in food processing and preparation. It is highly desirable to have a cost-effective, high-performance lubricant that is approved for such uses by the United States Department of Agriculture.

In addition, process conditions play a significant role in determining both the performance and commercial acceptance of a synthetic lubricant. If the heating conditions during preparation of the lubricant are not optimal, *e.g.*, if the lubricant is heated too much or too little or at the wrong point in the process, the lubricant can have an undesirable color or smell. One popular ingredient, Irgalube, can impart a sweet smell that is not always

desirable. However, omitting this ingredient to eliminate the odor can alter the performance characteristics of the lubricant. In addition, replacement with DuPont's Teflon® may result in a lubricant that has an uneven consistency. Replacement with Krylon® (liquid Teflon® in an isopropyl base) may resolve the consistency problems, but the isopropyl doesn't react well at high temperatures.

Summary of the Invention

The present invention relates to synthetic lubricant compositions and methods for their manufacture and use. The compositions comprise mixtures of hydrogenated poly-α-olefins, styrene-ethylene/propylene copolymer, petroleum hydrocarbons; fumed silica, propylene glycol, and PTFE.

Finally we got some PTFE which was a fine ground product and could easily be mixed into our lubricant. This not only solved the problem of the smell but, added a nice appearance to my product. Also, the grease performed at a much higher level then anything else that we tried.

The presently claimed synthetic lubricant compositions provide several unique performance advantages over known lubricants. The presently claimed compositions are either clear or translucent to white and non-staining. In addition, they have absolutely no odor, are non-toxic, and all ingredients in the grease are USDA approved. Finally, they retain their high-performance characteristics through a temperature range of about –40°F up to about 550°F.

Detailed Description

The present invention may be embodied in a variety of formulations. One feature of the invention, putting high heat on the poly-α-olefin oil, enables the consistent production of a high-quality composition; even if you overheat the batch, you still won't burn the oil.

The ranges of each component of the composition are as follows:

Ingredient	CAS Number	wt %
1.) Hydrogenated Poly-α-olefins	68037-01-4	33-81
2.) Styrene-Ethylene/Propylene Copolymer	68648-89-5	2-4
3.) Petroleum Hydrocarbons	8042-47-5	1-60
4.) Fumed Silica	112945-52-5	5-10
5.) Propylene Glycol	029434-03-5	2-5
6.) PTFE	79070-11-4	1-5

Optionally, 2-5 wt % polybutane (CAS No. 9003-29-6) may be added.

Example 1

A 10% by weight Shelvis solution was prepared using a 55 gallon drum with a high-temperature heat belt. 375 lbs of Amoco Dynacyn 168 (poly-α-olefin) was pre-heated to 200-240°F. 40 lbs Shelvis-50 (styrene-ethylene/propylene copolymer) powder was added to the pre-heated poly-α-olefin and the mixture was mixed under low agitation for approximately 3 hours with a 20 hp Schold mixer until the powder was melted. The liquid was strained through a 100 μ mesh strainer (filter bag).

Example 2

PTFE paste was prepared by adding 40 lbs of PTFE powder to 20 lbs Amoco Dynacyn 168 (poly-α-olefin) and grinding the mixture together at 140°F until it formed a paste.

Example 3

A synthetic high-temperature grease was prepared as follows. 110 lbs of the 10% Shelvis solution was mixed with 50 lbs Dynasyn 168 (poly-α-olefin), and 105 lbs mineral oil and the mixture was preheated to 125°F. 11 lbs Indepol 300 (polybutane), 8 lbs PTFE paste, 18 lbs fumed silica M-5, and 10 lbs propylene glycol 2025 were slowly sequentially added as the mixture was mixed. This yielded 312 lbs of the composition.

Example 4

Ingredient	CAS Number	wt %
1.) Hydrogenated poly-α-olefins	68037-01-4	48
2.) Styrene-Ethylene/Propylene Copolymer	68648-89-5	3.5
3.) Petroleum Hydrocarbons	8042-47-5	33
4.) Polybutane	9003-29-6	3.5
5.) Fumed Silica	112945-52-5	7
6.) Propylene Glycol	029434-03-5	3
7.) PTFE	79070-11-4	2

A high-temperature, synthetic lubricant composition was prepared as follows. Hydrogenated poly-α-olefins were heated to about 240°F. Styrene-ethylene/propylene copolymer was added and mixed at low agitation until dissolved (melted) and the solution was strained through a 100 micron mesh filter bag. Petroleum hydrocarbons and polybutane were slowly added and mixed for 15 minutes. PTFE was added and the composition mixed for 10 minutes. At this point, it is important to verify that the temperature of the composition is 125°F. Fumed silica was then slowly added, making sure not to create too much dust. Finally, polypropylene glycol was added and the composition was mixed thoroughly and run through a Cornell Versator.

* * *

While the present invention has been described in terms of specific methods and compositions, it is understood that variations and modifications will occur to those skilled in the art upon consideration of the present invention. Numerous modifications and variations in the invention as described in the above illustrative examples are expected to occur to those skilled in the art.

WHAT IS CLAIMED IS:

1. A high-temperature, synthetic lubricant composition comprising 33-81 wt % hydrogenated poly-α-olefins, 2-4 wt % styrene-ethylene/propylene copolymer, 1-60 wt % petroleum hydrocarbons, 5-10 wt % fumed silica, 2-5 wt % propylene glycol, and 1-5 wt % PTFE.

2. The high-temperature, synthetic lubricant composition as claimed in claim 1 provided in an atomizing dispenser selected from the group consisting of aerosol and non-aerosol dispensers.

3. The high-temperature, synthetic lubricant composition as claimed in claim 1 provided in a form selected from the group consisting of solid and semi-solid greases, pastes, and colloidal suspensions.

4. The high-temperature, synthetic lubricant composition as claimed in claim 1 further comprising 2-5 wt % polybutane.

5. The high-temperature, synthetic lubricant composition as claimed in claim 4 provided in an atomizing dispenser selected from the group consisting of aerosol and non-aerosol dispensers.

6. The high-temperature, synthetic lubricant composition as claimed in claim 4 provided in a form selected from the group consisting of solid and semi-solid greases, pastes, and colloidal suspensions.

7. A method for manufacturing a high-temperature, synthetic lubricant composition, the method comprising the following steps:

(a) heating 33-81 wt % hydrogenated poly-α-olefin to about 240°F;

(b) adding 2-4 wt % styrene-ethylene/propylene copolymer to said heated

hydrogenated poly-α-olefin;

(c) mixing the above ingredients until the styrene-ethylene/propylene copolymer is dissolved;

(d) straining the resultant solution;

(e) slowly adding 1-60 wt % petroleum hydrocarbons and further mixing the composition;

(f) adding 1-5 wt % PTFE and further mixing the composition;

(g) verifying that the temperature of the composition is about 125°F

(h) slowly adding 5-10 wt % fumed silica to the above mixture;

(i) Adding 2-5 wt % polypropylene glycol to the above mixture

(j) filtering the thoroughly mixed composition.

8. The method as claimed in claim 7 wherein the mixing in step (c) is conducted under low agitation.

9. The method as claimed in claim 7 wherein the straining in step (d) is accomplished through a 100 micron mesh filter bag.

10. The method as claimed in claim 7 wherein the mixing in step (e) is conducted for about 15 minutes.

11. The method as claimed in claim 7 wherein the mixing in step (e) is conducted under low agitation.

12. The method as claimed in claim 7 wherein the mixing in step (f) is conducted for about 10 minutes.

13. The method as claimed in claim 7 wherein the mixing in step (f) is conducted under low agitation.

14. The method as claimed in claim 7 wherein the addition in step (i) is conducted while mixing the composition under low agitation.

15. The method as claimed in claim 7 further comprising addition of 2-5 wt % polybutane in step (e).

16. The method as claimed in claim 15 wherein the mixing in steps (c), (e), and (f) are conducted under low agitation.

17. The method as claimed in claim 15 wherein the straining in step (d) is accomplished through a 100 micron mesh filter bag.

18. The method as claimed in claim 15 wherein the mixing in step (e) is conducted for about 15 minutes.

19. The method as claimed in claim 15 wherein the mixing in step (f) is conducted for about 10 minutes.

20. The method as claimed in claim 15 wherein the addition in step (i) is conducted while mixing the composition under low agitation.

synthetic multi-purpose, heavy duty grease specifications

Composition		Synthetic / Polymer
Visual Appearance		Optically Clear
Shelf Life		> 10 years
Oil Separation Test	ASTM D1742 60°C 24 Hours	Zero
	120°C 24 Hours	Zero
Viscosity	Brookfield at 25°C	1800 mcps +/- 100mcps
Cone Penetration	ASTM D217 (unworked)	
	at +25°C	240 +/- 20
	at -20°C	160 +/- 20
Drop Test	ASTM D566 at 250°C	Zero
Flash Point	ASTM D92	180°C
Continuous Use Temp.	Grease remains flexible & stable	-41°C to 204°C
Effect on Copper	ASTM D1261	Zero
Effect on plastic, steel iron, bronze, acrylate	Electron Microscopy Detection of deterioration	No evidence is observed
Volatility	150°C for 24 hours	< 1%
Evaporation Test	ASTM D972 85°C for 24 hrs.	< .3%
Oxidation Induction Time	Bellcore TR- TSY000421	> 20 Minutes
Water Content	Karl Fisher Method	< .001%
Hydrogen Generation	ASTM D1018	< 30 ppm
Four Ball Wear Test	ASTM D2266 Scar diameter	0.48 mm
Salt Spray Test	ASTM B117	Pass
Four Ball E.P. Test	ASTM D2596	
	Load Wear Index	43.25 kg.
	Weld Point	208 kg.
Water Washout	ASTM D1264	
	37.8°C (100°F)	< 1%
	79.4°C (175°F)	< 1%
Rust Preventative Test	ASTM D1743	Pass

FIG. 1

INTERNATIONAL SEARCH REPORT

In onal application No.
PCT/US01/14430

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :C10M 125/26, 169/04
US CL : 508/138
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 508/138

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST; WEST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,396,514 A (RANDISI) 02 August 1983	1-20
A	US 5,037,566 A (RANDISI) 06 August 1991	1-20

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

14 JUNE 2001

Date of mailing of the international search report

03 JUL 2001

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231
Facsimile No. (703) 305-3230

Authorized officer
JACQUELINE V. HOWARD
Telephone No. (703) 308-0661